Heavy Vehicle Propulsion Materials

Ultra-high Resolution Electron Microscopy for Catalyst Characterization

Background

The new aberration-corrected electron microscope (ACEM) is now in its first year of full beneficial operation. It has been employed in several studies to better understand the structures of catalyst materials for reduction of emissions of nitrous oxides in automotive and diesel exhaust systems. The ACEM is the first such microscope in the nation that is located in a user facility. It supports not only Energy Efficiency and Renewable Energy (EERE) Program catalyst studies, but also a variety of user projects that are related and that provide synergistic information to aid in the understanding of catalyst micro-structure, behavior, and poisoning mechanisms. Utilizing the "annular dark-field" or so-call "Z-contrast" imaging mode, the instrument provides sub-Ångström (1 Å = 0.1 nm) images that show high atomic number atomic species in bright contrast, directly related to relative atomic number. Thus, it is now possible to image single atoms, ultra-fine clusters, and "rafts" of atoms that are precursor structures in typical as-prepared catalyst systems.

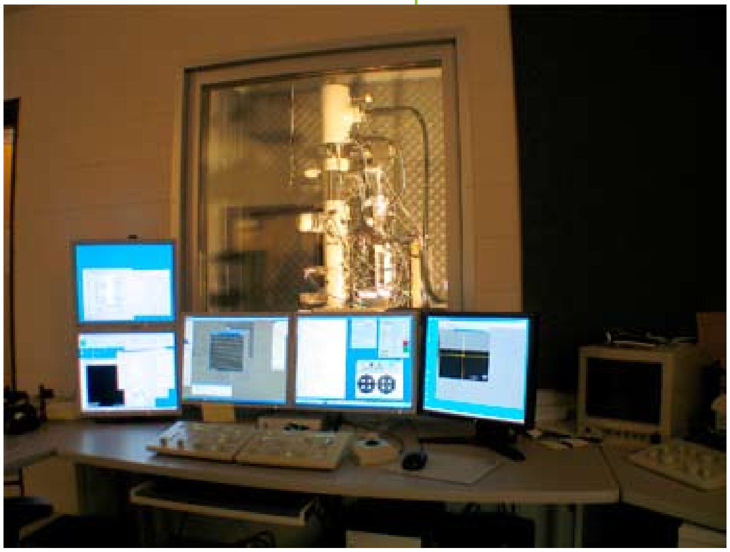

Figure 1. *JEOL 2200FS-AC aberration-corrected STEM/TEM, seen through a window in the control room. All alignments and other operations are controlled using an associated knobset, and mouse clicks within the digital imaging software.*

The goal of this research is to find effective methods to control the morphology of the catalytic during use so that optimum performance can be maintained. Outstanding results have been obtained recently in studies of rhenium and platinum catalysts on alumina support materials.

Benefits

- The most advanced catalyst imaging capability made available to DOE programs.
- Will further the understanding of the important mechanisms for catalyst deactivation with use.

Technology

To assure that the ACEM can achieve its optimum resolution on a routine basis, the instrument is housed in the new Advanced Microscopy Laboratory, located at the Oak Ridge National Laboratory (ORNL). This new facility was designed to provide an environmentally ‘quiet’ environment for ultra-sensitive instruments. The ACEM is operated remotely, from an adjacent control room, as shown in Figure 1. This capability also allows it to be accessed from any other remote location, which will greatly facilitate collaborative research with outside partners.

The ability of the ACEM to characterize catalyst microstructures at the atomic level is shown most spectacularly with the imaging of rhenium (Rh) clusters on γ-alumina. In this work with Prof. Bruce Gates of the University of California, Davis, Rh-carbonyl clusters containing only 3 Rh atoms were distributed on the surfaces of fine alumina support particles. The clusters cannot be detected in a bright-field image due to the high phase contrast of the support structure. In dark-field mode, however, the strongly scattered electron intensity from the Rh reveals the species in bright contrast. Figure 2 shows single atoms (A), a 3-atom trimer (B), and an apparent dimer (C). As indicated in Figure 3, the dimer shows double the intensity of one atom versus the other,

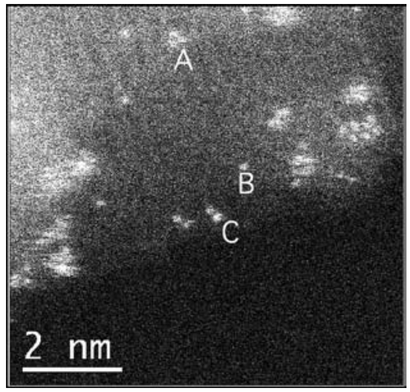

Figure 2. *Dark-field mode reveals rhenium clusters on alumina.*

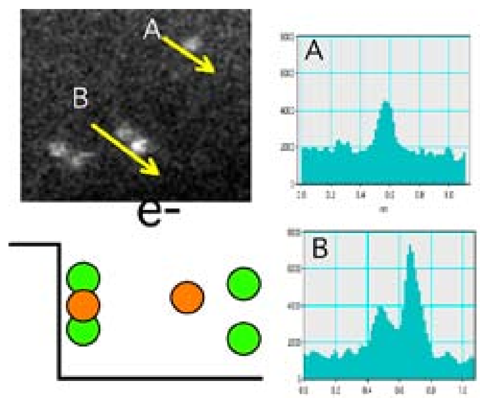

Figure 3. *Enlargement of apparent rhenium dimer reveals significant difference in intensity of atoms.*

suggesting that we are imaging a trimer edge-on, perhaps tethered to a ledge on the alumina surface, as indicated by the cartoon.

Status

This is the first time such a capability for catalyst characterization at the atomic level has been unambiguously demonstrated. This capability will be further utilized in advanced studies of catalysts of interest in the FCVT Program.

Contacts

Dr. Larry Allard
Oak Ridge National Laboratory
(865) 574-4981
allardlfjr@ornl.gov

Dr. James Eberhardt
DOE Technology Manager
Department of Energy
(202) 586-9837
james.eberhardt@ee.doe.gov

A Strong Energy Portfolio for a Strong America

Energy efficiency and clean, renewable energy will mean a stronger economy, a cleaner environment, and greater energy independence for America. Working with a wide array of state, community, industry, and university partners, the U.S. Department of Energy’s Office of Energy Efficiency and Renewable Energy invests in a diverse portfolio of energy technologies.